Performance Monitoring Protocol (QA/QC) for the Ion Chromatography (IC) System

1 Scope

This document addresses the performance monitoring (QA/QC) of the Ion Chromatography (IC) system. This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives (chemistry) examinations performed at the Huntsville facility.

2 Principle

The IC system is a high performance liquid chromatography (HPLC) pump and a conductivity detector. The instrument can be configured to analyze anions or cations. When an ion elutes from the column and passes the detector, it produces a change in conductivity which is recorded as a peak in the chromatogram.

This performance monitoring protocol is based upon the manufacturer's recommendations. The Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

3 Equipment/Materials/Reagents

a. Instrumentation - Dionex ICS-2100 or Dionex Integrion HPLC pump and suppressed conductivity detector, Dionex AS-AP programmable autosampler, and Chromeleon Software (or equivalent)

b. Instrumentation - Waters e2695 Separations module, Waters 2432 Conductivity Detector, and Empower Software (or equivalent)

c. Columns:
IonPac CS12A Analytical Column (Thermo Dionex or equivalent)
IC-Pak C M/D Analytical Column (Waters or equivalent)
IonPac AS19 Analytical Column (Thermo Dionex or equivalent)
IonPac AS22 Analytical Column (Thermo Dionex or equivalent)
IonPac AG19 Guard Column (Thermo Dionex or equivalent)
IonPac AG22 Guard Column (Thermo Dionex or equivalent)
IonPac CG12A Guard Column (Thermo Dionex or equivalent)

d. Nitric Acid (HNO_3) (Reagent Grade)

e. Ethylenediaminetetraacetic Acid (EDTA) (Reagent Grade)

f. Thermo Dionex EGC III or EGC 500 KOH RFIC Eluent Generator (Potassium Hydroxide) (or equivalent)

g. Thermo Dionex EGC III or EGC 500 MSA RFIC Eluent Generator (Methanesulfonic Acid) (or equivalent)

h. Thermo Dionex EGC 500 K_2CO_3 RFIC Eluent Generator (Potassium Carbonate) (or equivalent)

i. Deionized Water, 18.2 MΩ Milli-Q or equivalent

Redacted

l. Syringe - 250 µL (Thermo Dionex or equivalent)

4 Standards and Controls

4.1 Anions Testmix

The Testmix is used to assess daily operating performance and continued integrity of the system.

To prepare:

Pipette 5 mL of each liquid component of the anion standard for IC Redacted and 5 mg of each solid component Redacted into a 250 mL volumetric flask. Dilute to volume with deionized 18.2 MΩ water. The testmix will be maintained in a plastic bottle in the refrigerator. This preparation may be appropriately scaled.

4.2 Cations Testmix

The Testmix is used to assess daily operating performance and continued integrity of the system.

To prepare:

Pipette 5 mL of each cation standard for IC Redacted into a 250 mL volumetric flask, and dilute to volume with

deionized 18.2 MΩ water. The testmix will be maintained in a plastic bottle in the refrigerator. This preparation may be appropriately scaled.

5 Sampling

Not applicable.

6 Procedures

6.1 Daily Checks

The following steps will be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

a. Record the remaining disk space on the hard drive. Use Windows to verify that the hard disk has at least 100 MB of free space. Do not use if less than 100 MB remain.

b. Check the level of deionized water in the reservoir and make sure there is sufficient volume to complete the sequence.

c. Check the level of the waste container. Empty if necessary.

d. Perform an analysis of the appropriate Testmix. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the chromatogram.

e. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

6.2 As Needed Checks and Maintenance

a. Fill the needle wash reservoir (if applicable).

b. Replace the Eluent Generator.

c. Replace the guard column.

d. Replace the analytical column.

7 Instrumental Conditions

7.1 Thermo Dionex Cations

Mobile Phase:	Methanesulfonic acid (20 mM), supplied from Eluent Generator
Pump Mode:	Isocratic
Flow Rate:	1.0 mL/min
Column:	Thermo Dionex IonPac CS12A 4x250 mm with IonPac CG12A Guard 4x50 mm
Column Temperature:	30°C
Inj Volume:	25 µL
Acquire Time:	15 minute minimum

7.2 Waters Cations

Mobile Phase:	3.0 mM Nitric Acid (HNO_3) / 0.1 mM EDTA
Pump Mode:	Isocratic
Flow Rate:	1.0 mL/min
Column:	Waters IC-Pak Cation M/D 3.9x150 mm
Column Temperature:	Ambient
Inj Volume:	10 µL
Acquire Time:	15 minute minimum

7.3 Thermo Dionex Anions (Potassium Hydroxide Method)

Mobile Phase:	Potassium Hydroxide (gradient 20-80 mM)
Pump Mode:	Multi-step gradient (20 mM at 0 min, 20 mM at 2 min, 30 mM at 9 min, 80 mM at 13 min, 80 mM at 21min, 20 mM at 21.1 min, 20 mM at 25 min), supplied from Eluent Generator
Flow Rate:	1.0 mL/min
Column:	Thermo Dionex IonPac AS19 4x250 mm with IonPac AG19 Guard 4x50 mm
Column Temperature:	30°C
Cell Temperature:	35°C
Inj Volume:	25 µL
Acquire Time:	25 minute minimum
Suppressor:	Non-carbonate specific suppressed conductivity

7.4 Thermo Dionex Anions (Potassium Carbonate Method)

Mobile Phase:	Potassium Carbonate (10 mM), supplied from Eluent Generator
Pump Mode:	Isocratic
Flow Rate:	1.5 mL/min

Column:	Thermo Dionex IonPac AS22 4x250mm with IonPac AG22 Guard 4x50 mm
Column Temperature:	35°C
Cell Temperature:	35°C
Inj Volume:	25 µL
Acquire Time:	16 minute minimum
Suppressor:	Non-carbonate specific suppressed conductivity

8 Decision Criteria

Verify the results of the Testmix.

a. In order for the instrument to be considered in good operating condition, all components from the appropriate Testmix should generate well-resolved, Gaussian-shaped peaks with baseline separation.

b. A SNR of 3:1 will be the minimum response necessary to consider a response a peak.

c. The retention times of the appropriate Testmix components should not deviate by ±5% compared to the previous run of the appropriate Testmix.

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel shall perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal

protective equipment should be used when handling any chemical and when performing any type of analysis.

13 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (IOG 001) *Instrument Operations Group SOP Manual.*

"Liquid Chromatograph General Maintenance Protocol" (IOG 003) *Instrument Operations Group SOP Manual.*

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
0	10/04/18	New document that specifies instrument protocol for the Huntsville facility.
1	06/15/20	Updated "Scientific and Biometrics Analysis Unit" in header and approval section. Updated wording in Section 2. Added Redacted to Sections 3j and 4.1. Added Redacted to Sections 3k and 4.1 Added EGC 500 to Section 3g. Removed "Record stock solutions preparations in the Reagent Log" and Shelf life from Sections 4.1 and 4.2. Updated heading in Section 6.2. Removed sentence about minor deviations from Section 7. Added specific suppressor type to Sections 7.3 and 7.4. Added unit name to title of each approver.

Approval

Redacted - Signatures on File

Scientific and Biometrics Analysis Unit Chief _ Date: 07/14/2020

Explosives Unit-Chemistry Technical Leader _ Date: 07/14/2020

Explosives Unit Chief _ Date: 07/14/2020